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EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Ti-13Nb-13Zr ALLOY PRODUCED BY POWDER METALLURGY

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Abstract. Ti-13Nb-13Zr alloy produced via powder metallurgy was submitted to heat treatment under various conditions and the effects on microstructure and elastic modulus were investigated. Heat treatment was performed using temperatures above and below α/β transus combined with different cooling rates – furnace cooling and water quenching. Microstructure and phases were analyzed employing scanning electron microscopy and X-ray diffraction. Elastic Modulus was determined using a dynamic mechanical analyzer (DMA). The results indicated that α phase precipitation and elastic modulus values increased after heat treatment performed using temperature below α/β transus. However, when it was performed above α/β transus and using higher cooling rate, a decrease in elastic modulus was observed despite higher α phase precipitation, indicating that the microstructural modifications observed via SEM, due to the presence of martensitic α phase, influenced on elastic modulus values.

Introduction

Biomaterials are natural or artificial materials used on fabrication of structures or implants that replaces lost or damaged biological parts, restoring form and function improving quality and longevity of human life [1,2].

Among metallic biomaterials, the use of titanium and its alloys is continuously increasing due to their high strength-to-weight ratio, superior biocompatibility and corrosion resistance, good mechanical properties and lower elastic modulus [3].

The use of materials with low elastic modulus, equivalent to that of the bone, is an important aspect for the success of an implant, preventing the insufficient load transferring between bone and implant that results in bone resorption and implant loosening, which is called stress shielding effect [4,5]. The wear resistance of biomaterials is dependent on the kind of alloy and microstructures, and surface hardening is one of the most effective methods of improving the wear resistance of titanium alloys [6].

Heat treatment is an effective way of introducing microstructural modifications on Ti alloys, due to the dependence of α+β alloys on morphology of α and β phases and its correlation with mechanical properties [7,8]. In this work the effects of heat treatment performed above and below α/β transus temperature on microstructure and elastic modulus of Ti-13Nb-13Zr alloy produced via powder metallurgy were studied.

Experimental

Commercially pure (CP) titanium, niobium and zirconium were heat treated under hydrogen atmosphere of 1 MPa at 700, 600 and 500°C, respectively, and then mechanically broken in particles (<425 μm). The hydride powders were weighted, Ti - 74, Nb - 13, Zr - 13 (%wt) and milled in high energy planetary ball milling (HEPBM) using 200 rpm speed during 180 minutes.

Zirconium oxide grinding bowl and balls were used aiming the reduction of contaminant levels introduced during milling stage. The proportion used between grinding balls and material was 10:1 (in mass), and cyclohexane was used as organic additive. The milled powders were isostatically pressed at 200 MPa and sintered at 1150°C for 10 hours under high vacuum (10^{-3} Pa), followed by air cooling (AC).

Heat treatment was conducted on sintered samples using temperatures of 650 and 900°C during 1h under high vacuum (10^{-3} Pa), followed by furnace cooling (FC) and water quenching (WQ).

Microstructures and phases were characterized using scanning electron microscopy (SEM) and elastic modulus was determined using a dynamic mechanical analyzer (DMA), by means of three point bending flexural test.

Results

Fig. 1 shows SEM image of Ti-13Nb-13Zr alloy sintered at 1150°C for 10h followed by air cooling before heat treatment. Matrix is composed of β phase and α phase is present in grain in form of needles, referred as Widmanstätten pattern. Minor equiaxial grains containing exclusively α phase are also visible.

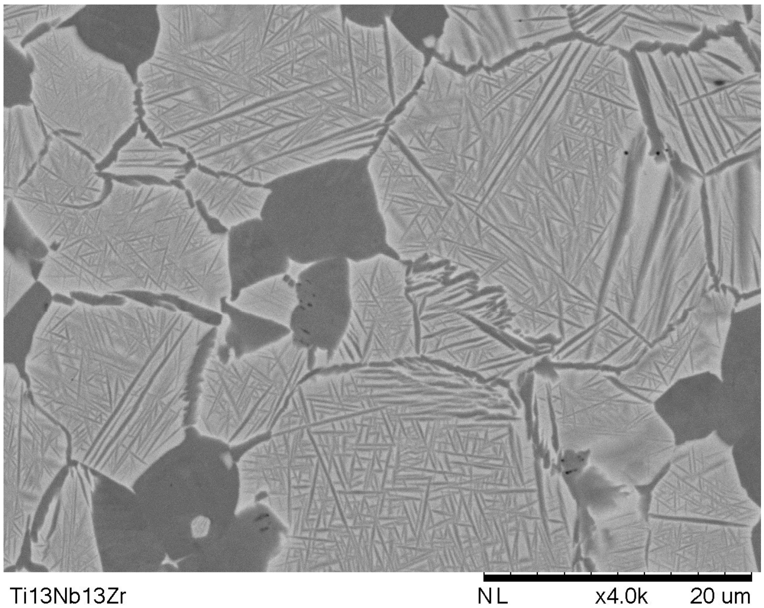

Fig. 1 – SEM images of Ti-13Nb-13Zr alloy produced by PM, sintered at 1150°C for 10h, using HEPBM with 200 rpm speed during 180 min followed by air cooling.

Fig. 2 (a-b) shows SEM image of Ti-13Nb-13Zr alloy sintered at 1150°C for 10h after heat treatment using temperature of 650°C during 1h followed by different cooling conditions (Furnace and Water). Noticeable alterations were introduced into alloy microstructure, where the grain size was reduced and it acquired a more irregular aspect, followed by an increase of α phase precipitation on grain boundaries. A decrease on size of α phase needles formed in intragranular areas was also observed, indicating the possibility of transformation of martensitic α phase to grain boundaries and equiaxial α phase due to preferential stability.

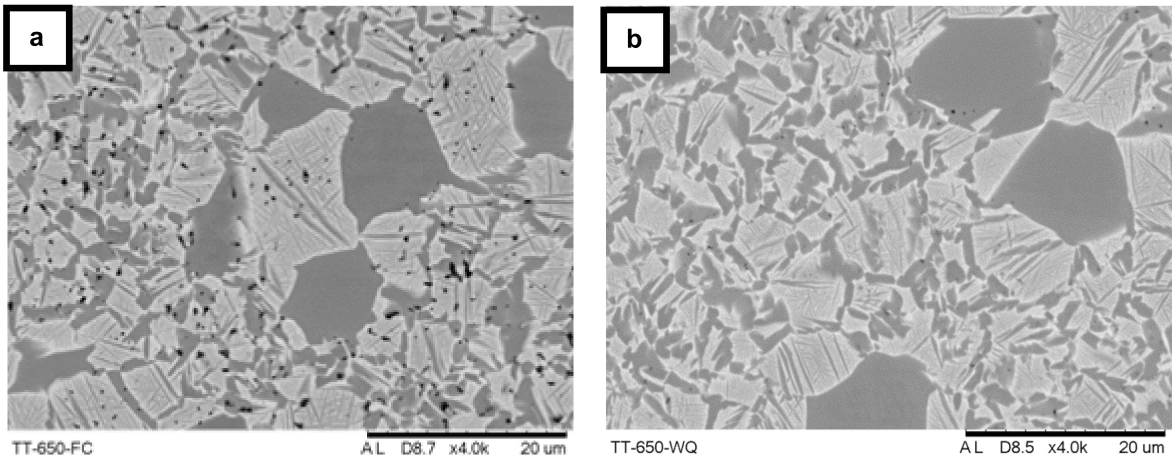

Fig. 2 – SEM images of Ti-13Nb-13Zr alloy sintered at 1150°C for 10h and submitted to heat treatment at 650°C during 1h, followed by (a) Furnace Cooling (FC), (b) Water Cooling (WQ)**.**

Fig. 3 (a-b) shows SEM image of Ti-13Nb-13Zr alloy sintered at 1150°C for 10h after heat treatment using temperature of 900°C during 1h followed by different cooling conditions. Using temperature above α/β transus and sufficiently slow cooling temperature (Fig. 3-a), the obtained microstructure was similar to heat treatment performed at 650°C. Using a faster cooling condition in water (Fig. 3-b), the microstructure was modified. Areas containing α phase on grain boundaries were increased and intragranular α phase showed an increase in thickness.

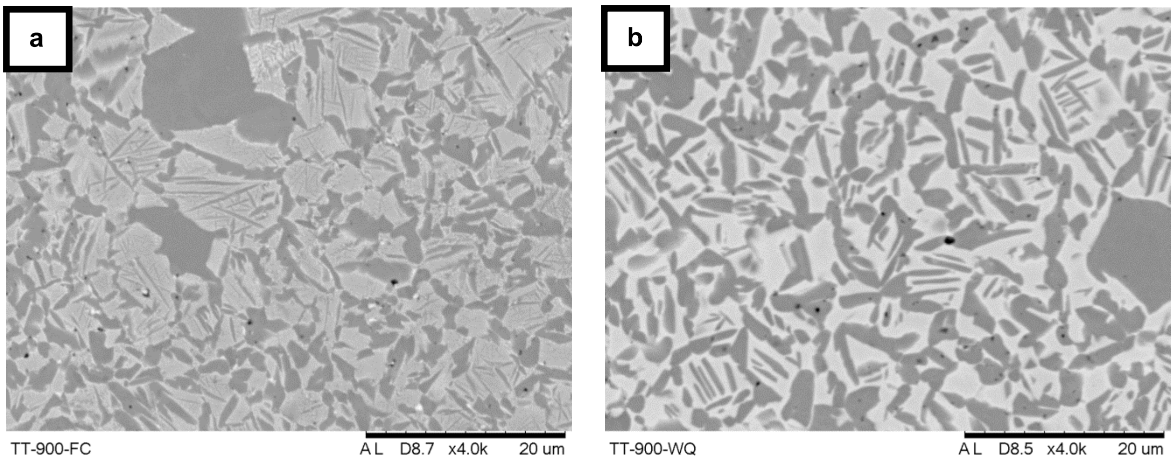

Fig. 3 – SEM images of Ti-13Nb-13Zr alloy sintered at 1150°C for 10h and submitted to heat treatment at 900°C during 1h, followed by (a) Furnace Cooling, (b) Water Cooling**.**

Fig. 4 shows X-Ray diffraction patterns of Ti-13Nb-13Zr alloy sintered at 1150°C for 10h after heat treatment under different conditions. When heat treatment was performed at 650°C, a reduction of relative intensity of 100% β phase corresponding peak was observed. Due to β phase metastability characteristics, the heat treatment acted as an aging treatment, increasing α phase precipitation.

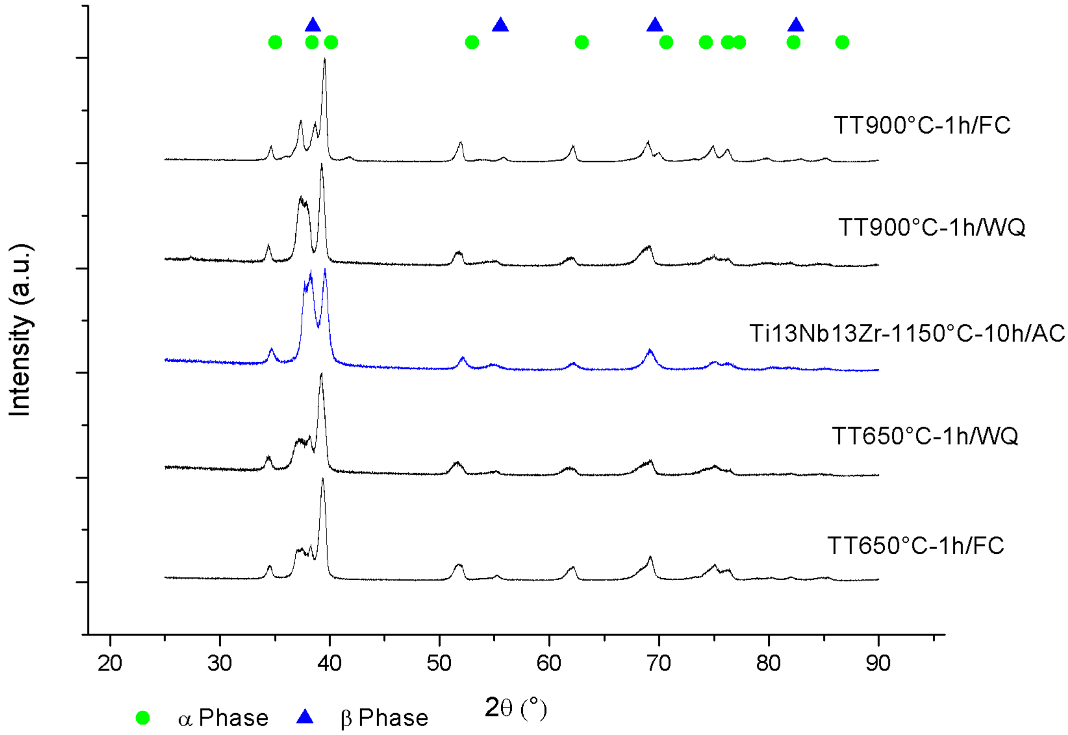

Fig. 4 – X-Ray diffraction patterns of Ti-13Nb-13Zr alloy produced by PM, sintered at 1150°C for 10h, followed by heat treatment under different conditions.

Elastic modulus of sintered Ti-13Nb-13Zr alloy after heat treatment is presented in Table 1. When the heat treatment was performed at 650°C, it produced an increase in elastic modulus when compared to initial sintered alloy, due to the higher precipitation of α phase, as observed in SEM images and X-Ray diffraction. Using 900°C temperature and a higher cooling rate (WQ), a small decrease in elastic modulus was observed, and it is associated to microstructural modification observed in SEM images and potential formation of martensitic α phase, which possess lower elastic modulus than regular α phase.

Table 1 – Elastic Modulus of Ti-13Nb-13Zr alloy submitted to heat treatment using temperatures above and below α/β transus.

Temperature (°C)	Elastic Modulus (GPa)	
	Furnace	Water
900	110.2	89.6
Initial Condition	91.7	
650	101.8	96.1

Conclusion

Heat treatment performed using temperatures above and below α/β transus temperature increased α phase precipitation and elastic modulus of studied materials, except for the condition using 900°C followed by WQ, where a small decrease of elastic modulus was observed due to microstructural modifications and potential formation of martensitic α phase.

Ti-13Nb-13Zr alloy sintered at 1150°C for 10h followed by heat treatment using 900°C and water cooling presented the most suitable properties for use as biomaterials.

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